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GYPSUM, HALOTRICHITE, AND SZOMOLNOKITE
FROM VINTON COUNTY, OHIO

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ABSTRACT

Samples of sulphate and carbonate efflorescences were collected for identification from several abandoned coal mine tunnels, cliff overhangs, and other sheltered sites along the Sandy Run Creek in Brown Township, Vinton County, Ohio.

These samples were analyzed optically and using x-ray powder diffractometer techniques in order to positively identify the minerals present.

Of the six samples analyzed, four proved to be gypsum, one contained both halotrichite, $\text{FeAl}_2(\text{SO}_4) \cdot 22\text{H}_2\text{O}$ and szomolnokite, $\text{FeSO}_4 \cdot \text{H}_2\text{O}$; and the last was determined to be calcite.

One of the sites visited during this study was examined earlier by Foster and Brant, who collected and identified magnesian halotrichite, but did not find szomolnokite (Foster and Brant, 1959).

The purpose of this paper is to present the results of the study done on the recent samples including identification data, locality descriptions, and a report on the processes involved in the deposition of these efflorescences.

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INTRODUCTION

This study was carried out in the Sandy Run Creek area of Vinton County because occurrences of halotrichite (Foster and Brant, 1959) and melanterite (Ehlers and Stiles, 1965) were previously reported to be found in two of the area's abandoned coal mine tunnels. On the basis of these reports, it seemed reasonable that other sulfate minerals occurring as efflorescences were likely to be found in a number of the area's mines since they too could provide the conditions required for the formation of such crusts.

SAMPLE LOCATION AND COLLECTION

The samples were collected from caves near three abandoned coal mine tunnels and from a nearby railroad tunnel. In each of the collection sites, the coal seam is the Middle Kittanning #6 Coal and the caves are formed from the overlying Lower Freeport Sandstone. These formations are of the Allegheny and Conemaugh Series of the Pennsylvanian System. The efflorescence occurred on the walls, ceilings, and floors of these sheltered areas and mine water was present in all sites except the railroad tunnel.

At the first sample location, a mine opening beneath an overhanging cliff, samples A-1 and A-2 were collected from the rear wall of the cave-like area by gently scraping the thin fragile crusts from about two feet above the coal seam, and six feet above the coal seam, respectively. Sample A-3, collected from this same site, was found on the cave floor, just above the water level in the mine. This specimen occurred as a mat of radiating aggregates of about one inch thickness that could be broken easily with a knife.

The next sample was collected from another mine opening, sheltered by a much smaller overhanging cliff, located about 200 feet north of the first site. Sample B was easily scraped from the bedrock at about six inches above the coal seam.

Another mine opening sheltered by a very large cliff overhang was located about 1700 feet farther north. Sample C was taken from the wall of this cave at about four feet above the coal seam and mine waters. This specimen consisted of a hard crust that was difficult to scrape from the bedrock.

The final sample, MT, was collected from the inside wall of the railroad tunnel at Moonville, two miles southeast of Lake Hope. The crusts found here

were thicker and less fragile than those found in the earlier-visited sites, but were still easily removed by scraping.

Each sample was stored in a sealable plastic bag to protect it from changes in humidity.



SCALE 1:36795



Taken from the Mineral Quadrangle, Ohio 7.5 Minute Series
(TOPOGRAPHIC)



Site A - Occurrences of gypsum, halotrichite and szomolnokite.



Sample A-3 Contains halotrichite and szomolnokite.



Site B - Occurrences of gypsum.



Site C - Occurrences of calcite.

SAMPLE ANALYSIS

Techniques

Each specimen was tentatively identified by examining its physical and optical properties both megascopically and microscopically.

Following this, each sample was analyzed using X-ray diffraction methods in order to positively identify the minerals present. The sample was powdered by grinding with a pestle and mortar and top-loaded into a sample holder for X-ray analysis. It was then run on a Norelco high angle diffractometer using copper K_{α} radiation and a nickle filter. Power was set at 35KV and 15ma. The samples were run through an arc of 4° to $60^{\circ} 2\theta$ and deflections were recorded on a strip chart. The observed diffraction patterns were then analyzed and the samples identified using the ASTM powder diffraction files.

Results

The data collected from both the optical examination and X-ray diffraction analysis shows that samples A-1, A-2, B, and M.T. are all gypsum.

In Sample A-3, optical and physical data indicates

the presence of halotrichite. X-ray diffraction data confirms this and suggests that another mineral is present. Since halotrichite has been previously reported to occur in association with szomolnokite at Quetena, Chile (Bandy, 1938) and at Dolliver State Park, Iowa (Cody and Biggs, 1973), the data was compared to the ASTM file on szomolnokite. The comparison led to the conclusion that the other mineral was, in fact, szomolnokite.

Optical and physical examination of Sample C suggests that this mineral is calcite. The identification is confirmed by the X-ray diffraction data.

The data on each sample is presented in the sections that follow.

SAMPLE OBSERVATIONS AND OPTICAL DATA

Samples A-1, A-2, B, and MT all occur as elongate to acicular crystals. These crystals have maximum size of 1cm and vary in color. Gypsum from Site A is yellow-green nearest the coal seam (A-1) and grayer in color toward the ceiling (A-2). The specimen from Site B is pink to gray. Sample MT is probably black due to coal soot inclusions. Microscopic examination provided more data on the minerals. Samples A-1, A-2, B, and MT are virtually identical. Each shows rhombohedral cleavage angles of 65° and 115° . All are colorless except MT, which contains inclusions of coal soot on the edges and ends of the crystals. Optically, all of the crystals are biaxially positive, maximum $Z\wedge C$ was found to be 51° , and all indices of refraction were estimated to be between 1.52 and 1.53 for each.

Sample A-3 occurs as acicular to hair-like crystals in mats of radiating aggregates. The crystals are white in color with a silky luster. Some aggregates are slightly stained yellow. Optical examination of the extremely thin crystals was limited to an approximate determination of the indices of refraction, which were found to be between 1.48 and 1.49.

The final sample, C, occurred as a hard, continuous, colorless to gray, surficial crust. Unlike the other crusts, it was smooth and microcrystalline. Microscopic examination of the extremely small crystals shows only that the mineral is uniaxial negative and that cleavage fragments are rhombohedral.

X-RAY DATA

Comparisons of the X-ray diffraction peaks given by the samples and the ASTM file are shown in the tables that follow.

COMPARISON OF X-RAY DATA FOR SAMPLES A-1, A-2, AND GYPSUM.

| SAMPLE A-1 | | SAMPLE A-2 | | GYPSUM * | |
|------------|------|------------|------|----------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 7.63 | H | 7.63 | H | 7.61 | 45 |
| 5.07 | L | 5.08 | L-M | | |
| 4.29 | H | 4.28 | H | 4.28 | 90 |
| 3.80 | H | 3.81 | H | 3.80 | 8 |
| | | 3.11 | L-M | | |
| 3.07 | H | 3.07 | H | 3.07 | 30 |
| 2.87 | M | 2.87 | M | 2.87 | 100 |
| 2.79 | L | 2.86 | L | 2.788 | 20 |
| 2.68 | M | 2.68 | M | 2.684 | 5 |
| 2.57 | L | 2.60 | L | 2.595 | 2 |
| 2.54 | L | 2.54 | L | | |
| 2.49 | L | 2.49 | L | 2.486 | 20 |
| 2.45 | L | 2.46 | L | 2.454 | 6 |
| 2.22 | L-M | 2.22 | L-M | 2.22 | 6 |
| | | | | 2.087 | 14 |
| 2.08 | L-M | 2.08 | L | 2.073 | 20 |
| 1.99 | L | 1.99 | L | 1.993 | 2 |
| 1.90 | L-M | 1.90 | M | 1.945 | 6 |
| 1.88 | L | 1.88 | L | 1.880 | 6 |
| | | 1.83 | L | | |

| SAMPLE A-1 | | SAMPLE A-2 | | GYPSUM | |
|------------|------|------------|------|--------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 1.81 | L | 1.81 | L | 1.812 | 4 |
| | | 1.78 | L | | |
| 1.76 | L | 1.77 | L | 1.778 | 4 |
| 1.67 | L | | | 1.685 | 2 |
| 1.62 | L | 1.62 | | 1.662 | 4 |
| 1.58 | L | 1.58 | L | 1.587 | 2 |

& additional
lines

*no lines omitted with intensities greater than 5

COMPARISON OF X-RAY DATA FOR SAMPLE A-3, HALOTRICHITE, AND SZOMOLNOKITE.

| SAMPLE A-3 | | HALOTRICHITE * | | SZOMOLNOKITE * | |
|------------|------|----------------|------|----------------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 9.51 | L | 9.69 | 20 | | |
| 7.87 | L | 7.92 | 15 | | |
| 6.03 | L-M | 6.10 | 20 | | |
| 5.83 | L | 5.81 | 10 | | |
| 5.27 | L | 5.25 | 10 | | |
| 4.95 | M | 4.97 | 50 | | |
| 4.86 | M | | | 4.85 | 30 |
| 4.80 | H | 4.81 | 100 | 4.79 | 16 |
| 4.67 | L | | | | |
| 4.60 | M | 4.63 | 30 | | |
| 4.36 | L | | | | |
| 4.30 | M | 4.30 | 100 | | |
| 4.15 | M | 4.15 | 50 | | |
| 4.11 | M | 4.10 | 50 | | |
| 3.95 | M | 3.98 | 20 | | |
| 3.77 | M | 3.78 | 30 | 3.73 | 6 |
| 3.61 | L | 3.66 | 15 | | |
| 3.50 | H | 3.50 | 100 | | |
| 3.44 | L | 3.40 | 25 | 3.44 | 100 |
| 3.33 | L | | | 3.37 | 25 |
| 3.27 | L | 3.27 | 25 | 3.28 | 25 |

| SAMPLE A-3 | | HALOTRICHITE | | SZOMOLNOKITE | |
|------------|------|--------------|------|--------------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 3.15 | L | 3.18 | 20 | 3.12 | 40 |
| 3.04 | L | 3.06 | 10 | | |
| 3.02 | L | 3.04 | 20 | | |
| 2.96 | L | 2.97 | 20 | | |
| 2.89 | L | 2.882 | 30 | | |
| 2.83 | L | 2.834 | 15 | | |
| 2.77 | L | 2.766 | 15 | | |
| 2.76 | L | | | | |
| 2.72 | L | 2.710 | 15 | | |
| 2.68 | L-M | 2.680 | 25 | | |
| 2.60 | L | 2.605 | 25 | | |
| 2.58 | L | 2.556 | 30 | | |
| 2.55 | L | 2.512 | 10 | 2.521 | 35 |
| 2.46 | L | 2.458 | 15 | 2.432 | 6 |
| | | 2.436 | 15 | | |
| 2.40 | L | 2.389 | 15 | 2.394 | 4 |
| 2.28 | L | 2.289 | 20 | 2.326 | 10 |
| | | | | 2.310 | 8 |
| 2.23 | L | 2.232 | 10 | 2.242 | 16 |
| | | 2.191 | 10 | 2.103 | 10 |
| 2.02 | L | | | 2.080 | 20 |

& additional
lines

* no lines omitted with intensities greater than 5

COMPARISON OF X-RAY DATA FOR SAMPLES B, MT, AND GYPSUM.

| SAMPLE B | | SAMPLE MT | | GYPSUM * | |
|----------|------|-----------|------|----------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 7.64 | H | 7.63 | H | 7.61 | 45 |
| 7.07 | L | | | | |
| 4.29 | H | 4.28 | H | 4.28 | 90 |
| 3.80 | H | 3.80 | H | 3.80 | 8 |
| 3.34 | H | 3.34 | H | | |
| 3.18 | L | 3.18 | L | 3.17 | 4 |
| 3.07 | H | 3.06 | H | 3.07 | 30 |
| 2.87 | M | 2.87 | M | 2.87 | 100 |
| 2.79 | L | 2.78 | L | 2.788 | 20 |
| 2.68 | M | 2.68 | M | 2.684 | 5 |
| 2.60 | L | 2.59 | L | 2.595 | 2 |
| 2.53 | L | 2.53 | L | 2.486 | 20 |
| 2.58 | L | | | 2.475 | 2 |
| 2.46 | L | | | 2.454 | 6 |
| 2.22 | L-M | 2.22 | L-M | 2.220 | 6 |
| 2.09 | L | 2.09 | L-M | 2.087 | 14 |
| 2.08 | L-M | 2.08 | L-M | 2.073 | 20 |
| | | 2.05 | L | 2.048 | 4 |
| 1.99 | L | 1.99 | L | 1.993 | 2 |
| | | 1.97 | L | 1.945 | 6 |
| 1.90 | L-M | 1.90 | L-M | 1.900 | 4 |
| 1.88 | L | 1.88 | L | 1.880 | 6 |

| SAMPLE B | | SAMPLE MT | | GYPSUM | |
|----------|------|-----------|------|--------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 1.81 | L | 1.81 | L | 1.812 | 4 |
| | | 1.80 | L | 1.798 | 6 |
| 1.78 | L | 1.78 | L | 1.778 | 4 |
| 1.62 | L | 1.62 | L | 1.622 | 4 |

& additional
lines

* no lines omitted with intensities greater than 5

COMPARISON OF X-RAY DATA FOR SAMPLE C, CALCITE, AND QUARTZ.

| SAMPLE C | | CALCITE * | | QUARTZ * | |
|----------|------|--------------------|------|--------------------|------|
| dÅ | I/I° | dÅ | I/I° | dÅ | I/I° |
| 4.26 | L-M | | | 4.26 | 35 |
| 3.85 | M | 3.85 | 26 | | |
| 3.35 | H | | | 3.343 | 100 |
| 3.03 | H | 3.03 | 100 | | |
| 2.84 | L | 2.834 | 2 | | |
| 2.50 | M | 2.495 | 7 | 2.458 | 12 |
| 2.28 | M | 2.284 | 21 | 2.282 | 12 |
| 2.09 | M | 2.094 | 32 | 2.128 | 9 |
| 1.93 | L | 1.926 | 4 | 1.980 | 6 |
| 1.91 | M | 1.9071 | 21 | | |
| 1.88 | M | 1.8726 | 42 | | |
| 1.82 | L | | | 1.817 | 17 |
| 1.62 | L | 1.6259 | 3 | | |
| 1.60 | L-M | 1.6040 | 21 | 1.608 | <1 |
| 1.54 | L | 1.5821 | 2 | 1.541 | 15 |
| 1.44 | L-M | | | 1.453 | 3 |
| | | & additional lines | | & additional lines | |

* calcite - no lines omitted

* quartz - no lines with intensities greater than 7 omitted

CONCLUSION

All of the sample sites are located in a geologic setting that provides an ideal environment for the formation of sulfate crusts. Materials needed for their formation are provided by the weathering of the abundant pyrite and marcasite in the coal seam which causes iron-staining and produces a high concentration of sulfuric acid in effluent mine waters. Groundwater percolating down through the porous sandstone provides more dissolved materials including calcium, magnesium, iron, and alumina.

These ions combine to form salts when this water evaporates after flowing directly onto a surface just above mine water level, or after capillary action draws the water out onto bedrock surfaces. The caves formed by the sandstone then prevent the dissolution of the salts by rain water.

The halotrichite and szomolnokite were found on sandstone just above the mine water level and their deposition probably depended on the high sulfuric acid content of these waters. In contrast, the gypsum and calcite occur on the sheltering sandstone and their deposition was probably dependent on the evaporation of

groundwater drawn onto the rock surface by capillary action. Similar processes are probably involved in the deposition of gypsum in the Moonville railroad tunnel. The same coal seam is present here but its elevation is at about the level of the tunnel's roof. Here, the groundwater transports the aqueous solution of calcium sulfate to the walls of the tunnel and gypsum is deposited upon evaporation of the groundwater. The origination of the gypsum crusts in the cracks between the bricks is one indication of this mode of deposition.

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